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STANDARDS OF PERFORMANCE FOR NEW STATIONARY SOURCES

Petroleum Refinery Sulfur Recovery Plants

ENVIRONMENTAL PROTECTION AGENCY

[40 CFR Part 60]

[FRL 610-7]

STANDARDS OF PERFORMANCE FOR NEW STATIONARY SOURCES

Petroleum Refinery Sulfur Recovery Plants

Notice is hereby given that under the authority of section 111 of the Clean Air Act, as amended, the Administrator is proposing standards of performance for new, modified or reconstructed petroleum refinery sulfur recovery plants.

The Administrator is also proposing a new reference method to be used for determining emissions of hydrogen sulfide and reduced sulfur compounds from petroleum refinery sulfur recovery plants.

PROPOSED STANDARDS

As proposed, the standards would apply to petroluem refinery sulfur recovery plants. Generally, these plants are physically located within petroleum refineries. Occasionally, however, refineries will pipe the sulfur-rich gas stream to a nearby facility, such as a chemical plant, where the sulfur is recovered. The standards would apply to all refinery sulfur recovery plants, whether they are physically located within a petroleum refinery, or within the confines of another facility.

The standards would limit the concentration of sufur dioxide (SO_2) in the gases discharged to the atmosphere to 0.025 pereent by volume at zero percent oxygen and on a dry basis. Where the emission control system installed to comply with these standards discharges residual emissions of hydrogen sulfide (H_2S), carbonyl sulfide (COS), and carbon disulfide (CS_2), the standards would limit the concentration of H_2S and the total concentration of H_2S, COS and CS_2 (calculated as SO_2) in the gases discharged to the atmosphere, to 0.0010 percent and 0.030 percent by volume at zero percent oxygen and on a dry basis, respectively.

ENVIRONMENT AND ECONOMIC IMPACTS

The proposed standards would require new, modified or reconstructed refinery sulfur recovery plants to reduce emissions by about 99 percent compared to emissions from existing refinery sulfur recovery plants. This would increase the overall sulfur recovery of a typical refinery sulfur recovery plant from about 94 percent to about 99.9 percent.

It is expected that the proposed standards would apply to about 80 new refinery sulfur plants by 1980. Few, if any, modifications or reconstructions of existing refinery sulfur plants are anticipated. The proposed standards would reduce national sulfur dioxide emissions from these new refinery sulfur plants by about 55,000 tons per year.

This reduction in national SO_2 emissions would be obtained without adverse impacts on other aspects of the environment, such as increased solid waste disposal, water pollution, or noise. The proposed standards would also result in a reduction in the growth of national energy consumption by some 54 million kwhr/yr, or about 90,000 barrels of fuel oil per year, by 1980.

The economic impact of the proposed standards would be reasonable both on large and on small refiners, although it is somewhat more severe on small refiners than large refiners. For the small refiner [processing less than 30,000 barrels per calendar day (BCD)], the proposed standards could reduce profitability, as measured by return on assets, by 1.5 to 7.5 percent. A price increase of only 0.25 to 1.0 percent on all petroleum products, however, would restore the small refiner's profitability. The impact on a large refiner (processing more than 30,000 BCD) is considerably less due to economies of scale. The proposed standards could reduce the profitability of a large refiner by 0.5 to 1.5 percent. A price increase of only 0.10 to 0.30 percent on all petroleum products, however, would restore the large refiner's profitability. Consequently, the proposed standards would not adversely affect the growth of either the large refinery or the small refiner sector of the domestic refining industry.

To comply with these proposed standards, the investment required by the domestic refining industry would total some $110 million (MM) over the five-year period from 1976 to 1980; the annual operating costs of the industry would increase by some $16 MM per year by 1980 and petroleum product prices could increase by 0.1 to 1.0 percent.

The review of the economic impact has shown that the proposal is not a major action under the Inflationary Impact Statement (IIS) program and no IIS is needed.

SELECTION OF SOURCE CATEGORY AND AFFECTED FACILITIES

Section 111 of the Clean Air Act directs the Administrator to establish standards of performance for new stationary sources of air pollution which may contribute significantly to air pollution which causes or contributes to the endangerment of public health or welfare. Sulfur recovery plants are a major source of SO_2 emissions in petroleum refineries. Although a few State or local regulations already require emission control equivalent to the proposed standards, most State regulations require less stringent control of refinery sulfur recovery plants.

Reducing SO_2 emissions from new, modified or reconstructed refinery sulfur recovery plants takes on importance in view of the expected growth in the numbers and sizes of these plants, and the urban location of many petroleum refineries where high ambient air concentrations of SO_2 already exist. Current growth projections for the domestic petroleum refining industry, for example, indicate that refinery sulfur recovery plant capacity will double between 1976 and 1980.

Considering these factors, the proposed standards have been developed to limit emissions of SO_2 from petroleum refinery sulfur recovery plants. Some of the emission control systems that might be installed to reduce emissions of SO_2, however, discharge residual emissions of hydrogen sulfide (H_2S), carbonyl sulfide (COS) and carbon disulfide (CS_2) to the atmosphere. The proposed standards, therefore, include limits on emissions of these pollutants.

RATIONALE

Two alternative systems of SO_2 emission control were considered as candiates to serve as the basis for standards of performance: (1) the low-temperature extended Claus reaction system (alternative I) and (2) various tail gas scrubbing systems (alternative II). The alternative I systems reduce emisssion from uncontrolled refinery sulfur recovery plants by 80–85 percent, while the alternative II systems reduce emissions by 98–99 percent. Overall sulfur recovery of the refinery sulfur plant is increased from 95 to 99.9 percent with the use of an alternative II system.

Standards of performance based on alternative I would have an essentially negligible impact on SO_2 emissions since most state implementation plans (SIP's) already require equivalent control. As outlined above, however, standards based on alternative II would reduce SO_2 emissions by about 90 percent below levels required by most SIP's and would lead to a reduction in national SO_2 emissions of some 55,000 tons per year in 1980. Standards based on alternative II would also have no adverse environmental impacts in other areas such as increased water pollution, solid waste disposal, energy consumption, or noise. In addition, the economic impact of standards based on alternative II would be reasonable both on large and on small refiners. Consequently, alternative II represents the best system of emission reduction and the proposed standards are based on this alternative.

The tail gas scrubbing systems of alternative II, however, include two different types of emission control systems for reducing SO_2 emissions from refinery sulfur recovery plants: oxidation control systems and reduction control systems. In the oxidation control system, the SO_2 emissions from a refinery sulfur plant are controlled directly by tail gas scrubbing. In the reduction control system, the SO_2 emissions are first converted to H_2S emissions which are then controlled by tail gas scrubbing. Residual emissions discharged into the atmosphere from oxidation control systems or from reduction control systems which are followed by incineration consist of SO_2. Residual emissions discharged from reduction control systems which are not followed by incineration, however, consist of H_2S, COS (carbonyl sulfide) and CS_2 (carbon disulfide). Standards limiting emissions of SO_2, therefore, while appropriate for oxidation control systems or reduction control systems followed by incineration, are not appropriate for reduction control systems not followed by incineration.

Where reduction control systems which are not followed by incineration

are properly operated and maintained, emissions of reduced sulfur compounds (H_2S, COS and CS_2) are insignificant. With poor operation and maintenance, however, emissions of these pollutants from these systems could lead to significant odor problems, primarily from the H_2S emitted. This situation presents a difficulty in selecting which pollutants should be controlled under the proposed standards due to the provisions of section 111(d) of the Act. Sulfur dioxide is a criteria pollutant for which State control of existing sources is not required under section 111(d) (since such control is already required by section 110). Reduced sulfur compounds, on the other hand, would be considered designated pollutants subject to the provisions of section 111(d).

Under the EPA regulations implementing section 111(d), after promulgation of a standard of performance for the control of such a designated pollutant, EPA must develop a guideline document containing the necessary information for States to develop plans for controlling emissions of the pollutant (40 CFR 60.22). In this case the document would have to cover information pertinent to the control of reduced sulfur compounds from all existing petroleum refinery sulfur plants which have installed reduction control systems which are not followed by incineration. After final issuance of the guideline document by EPA, all States containing affected plants are required by 40 CFR 60.23 to develop such plans and submit them to EPA for approval. Currently, about 25 refinery sulfur plants in seven States have installed reduction control systems which are not followed by incineration. Developing the guideline document and State plans, therefore, could be a significant undertaking, requiring the expenditure of considerable resources at the Federal level and by seven States and their local governments. Thus, one purpose of this proposal is to solicit comments from the affected States as to the burden imposed by the implementation of section 111(d) in this case.

If that burden imposed by the implementation of section 111(d) is great, EPA will consider the course of declining to require the adoption and submittal of State plans under 40 CFR 60.24. This option is available due to the unusual factual situation: all existing refinery sulfur plant, which have installed reduction control systems which are not followed by incineration (25 plants in seven States) already have very low emissions of reduced sulfur compounds. Their emission control systems have been installed to comply with State or local air pollution control regulations limiting emissions of SO_2 from existing refinery sulfur plants. To ensure that the installation of these emission control systems do not lead to local odor problems, these regulations also limit emissions of reduced sulfur compounds either directly or indirectly. Where emissions of reduced sulfur compounds are limited directly, local regulations specify the maximum concentrations of H_2S and reduced sulfur compounds (H_2S, COS and CS_2) that can be present in the tail gases discharged to the atmosphere. Where this approach has been followed, emissions of H_2S are limted to 10 ppm and emissions of reduced sulfur compounds are limited to either 300 or 500 ppm.

Where emissions of reduced sulfur compounds are limited indirectly by local regulations, these regulations require that the best available emission control technology be installed. In the process of specifying the best emission control technology, local air pollution control agencies have contacted EPA, vendors of various emission control system, and other local air pollution control agencies where these emission control systems have been installed. In terms of limiting emissions of reduced sulfur compounds, this approach has achieved the same end result as the direct regulatory approach. All 25 reduction control system which are now operating have been designed and guaranteed by the vendors of these systems to limit emissions to less than 10 ppm H_2S and less than 300 or 500 ppm reduced sulfur compounds.

Consequently, the current 25 petroleum refinery sulfur plants which have installed reduction control systems which are not followed by incineration are not considered by EPA to be significant sources of reduced sulfur compound emissions. Developing State plans under section 111(d) to control emissions of these pollutants from these facilities, therefore, would not reduce existing emission levels.

On the other hand, if EPA were to propose standards of performance for refinery sulfur plants only for SO_2 without limiting emissions of reduced sulfur compounds, this action could be interpreted to mean that EPA does not consider the potential air pollution problem posed by emissions of reduced sulfur compounds to be important. Such an interpretation could encourage new plants to be built without limiting emissions of these pollutants.

Having determined that the proposed regulation should cover emissions of reduced sulfur compounds from new refinery sulfur recovery plants, but not existing ones, EPA is left with the problem of selecting the appropriate pollutants to control with the proposed standards. Limiting emissions of SO_2 alone would be unsatisfactory due to the risk of reduced sulfur compound pollution from new plants. Limiting reduced sulfur compound emissions as well as SO_2 for new sources will cause some expenditure of effort on the part of EPA and the affected States which is not in fact necessary to achieve control of existing plants.

Considering all the facets of this situation, EPA proposes to limit emissions of both SO_2 and reduced sulfur compounds by these standards of performance, fulfilling the intent of section 111 of the Act to prevent new air pollution problems by requiring new, modified, or reconstructed plants to install the best systems of emission reduction considering costs. However, EPA is considering two alternative routes for treating the problem of existing sources. One alternative is for EPA to issue a guideline document under 40 CFR 60.22 (based primarily on the information available in the support documents for these standards of performance) and to require the seven affected States to submit plans under 40 CFR 60.23. Those States that already regulate reduced sulfur compounds by State law will be able to submit relatively simple control plans. Those States in which regulation of reduced sulfur compounds has been accomplished by local ordinances may have to provide for State enforcement before their control plans can be approved.

If the first alternative appears to be excessively burdensome, EPA is also considering a second alternative, because the 25 existing sources are well controlled. The purpose of limiting pollutant emissions from existing sources under the provisions of section 111(d) is to control a pollutant which may cause or contribute to the endangerment of public health or welfare, but is not known to be "hazardous" within the meaning of section 112, and is not controlled under sections 108 through 110. Since action under section 111(d) in this instance would not further control the pollutant in question, and existing refinery sulfur plants which have installed reduction control systems not followed by incineration are not endangering the public health or welfare, the conclusion can be drawn that there is no purpose to limiting emissions of these pollutants under section 111(d). Under this alternative, EPA would not issue a guideline document, the chain of events requiring States to develop plans for controlling emissions of these pollutants would not be initiated and these plans would not be developed. (A detailed discussion of this alternative may be found in the Standards Support and Environmental Impact Statement for Petroleum Sulfur Recovery Plants.)

The data and information available for selecting specific emission limits include the results of emission tests both by the Agency and by local air pollution control agencies, discussions with owners and operators which have installed and are now operating either the oxidation control systems or the reduction control systems, and discussions with the vendors of these emission control systems. The SO_2 emission limit is based on emission tests at a refinery sulfur plant controlled by an oxidation control system and at a refinery sulfur plant controlled by a reduction control system followed by incineration. The H_2S and reduced sulfur compound emission limits are based on emission source tests at three refinery sulfur plants controlled by reduction control systems not followed by incineration. The numerical values were selected including a reasonable margin for error taking into consideration the limited data base, the relative "newness" of the plants

tested, pilot-plant data available from vendors on long-term operation, and the experience of owners and operators with these emission control systems.

The 0.025 percent by volume limit for SO_2 emissions and the 0.030 percent by volume limit for reduced sulfur compound emissions are also equivalent, in the sense that both require achieving the same degree of overall sulfur recovery (i.e., 99.9 percent). The numerical limit on SO_2 emissions is lower due to the somewhat greater volume of gases discharged from oxidation control systems and reduction control systems followed by incineration, which is inherent in their design and operation.

It should be noted that standards of performance for new sources established under section 111 of the Clean Air Act reflect emission limits achievable with the best adequately demonstrated system of emission reduction considering the cost of such systems. State implementation plans (SIP's) approved or promulgated under section 110 of the Act, on the other hand, must provide for the attainment and maintenance of national ambient air quality standards (NAAQS) designed to protect public health and welfare. For that purpose SIP's must in some cases require greater emission reductions than those required by standards of performance for new sources. Indeed, building of new sources may be precluded altogether in some geographical areas. In addition, States are free under section 116 of the Act to establish more stringent emission limits than those established under section 111 or those necessary to attain or maintain the NAAQ under section 110. Thus, new sources may in some cases be subject to limitations more stringent than EPA's standards of performance under section 111, and prospective owners and operators of new sources should be aware of this possibility in planning for such facilities.

Energy

The impact of the proposed standards on the energy consumption of a refinery sulfur recovery plant varies depending on the type of emission control system installed to comply with the standards. If an oxidation control system or a reduction control system followed with incineration were used, the overall energy consumption of a typical sulfur recovery plant would be increased by some 30 percent. If a reduction control system not followed by incineration were used, however, the overall energy consumption would be reduced by some 50 percent. These percentages are high primarily because refinery sulfur recovery plants consume little energy. Typically, for example, a refinery sulfur recovery plant accounts for less than 1 percent of the total energy consumed within a petroleum refinery.

Assuming half of the refinery sulfur recovery plants affected by the proposed standards by 1980 install oxidation control systems or reduction control systems followed by incineration and half install reduction control systems not followed by incineration, the proposed standards would reduce national energy consumption in 1980 by some 54 million kw-hr/yr, or about 90,000 barrels of fuel oil per year.

Monitoring, Testing, and Recordkeeping

As proposed, the regulations would require monitoring of emissions released to the atmosphere to insure that the emission control systems installed to comply with the proposed standards are properly maintained and operated. If an oxidation control system or a reduction control system followed by incineration were installed, monitoring of SO_2 emissions would be required and if a reduction control system not followed by incineration were installed, monitoring of H_2S and reduced sulfur compound emissions would be required.

Emission monitoring systems for SO_2 are readily available and performance specifications for these monitors have already been promulgated by EPA in 40 CFR Part 60 Appendix B. Although a few emission monitoring systems for H_2S and reduced sulfur compounds are available, EPA has not yet developed performance specifications for these monitors. Consequently, until EPA proposes and promulgates these performance specifications, owners and operators subject to the requirement to install H_2S and reduced sulfur compound emission monitors will not be required to do so. The requirement to install these monitors is included in the proposed regulations; however, to ensure that when EPA promulgates these performance specifications, those owners and operators who have installed reduction control systems not followed by incineration which are subject to the proposed standards will be required to retrofit these monitors. This will enable enforcement personnel to ensure that these emission control systems are being properly operated and maintained.

The reference method for determining compliance also depends on the emission control system employed to comply with the proposed standards. If an oxidation control system or a reduction control system followed by incineration were installed, Method 6—Determination of Sulfur Dioxide Emissions from Stationary Sources would be used to determine emissions of SO_2. If a reduction control system not followed by incineration were installed, the proposed Reference Method 15—Determination of Hydrogen Sulfide and Reduced Sulfur Compound Emissions from Sulfur Recovery Plants would be used to determine emissions of H_2S and total emissions of H_2S, COS, and CS_2 (calculated as SO_2).

The proposed Reference Method 15 is based on gas chromatographic separation followed by flame photometric detection. This method permits separation and identification of each individual pollutant (i.e., H_2S, COS and CS_2). To determine compliance with the proposed standards, a sample of the gases discharged to the atmosphere is analyzed to determine emissions of H_2S, COS and CS_2. The concentration of H_2S in these gases is determined directly. The concentration of reduced sulfur compounds in these gases is determined indirectly, by adding together the concentrations of H_2S, COS and CS_2. Since emissions are calculated as SO_2, however, the concentrations of CS_2 (having twice the sulfur per molecule of gas) is multiplied by two before adding it to the concentrations of H_2S and COS.

Amendment of Subpart J of Part 60

The proposed standards would be incorporated into 40 CFR Part 60 by amending Subpart J—Standards of Performance for Petroleum Refineries. Maintaining clarity within Subpart J while incorporating the proposed standards requires amendment of a number of existing paragraphs. In all but the case discussed below, these paragraphs have merely been rewritten with no change in content or intent.

Recently, it has been brought to the attention of the Administrator that as Subpart J is now written, a fluid catalytic cracking unit incinerator waste-heat boiler is an affected facility subject to the standard limiting emissions of particulate matter. Thus, if an owner or operator installs an incinerator waste-heat boiler, that boiler must comply with the standard on particulate emissions.

An incinerator waste-heat boiler, however, is an emission control device which reduces emissions of carbon monoxide from a fluid catalytic cracking unit catalyst regenerator. It was not EPA's intent that installation of an emission control device to reduce carbon monoxide emissions would then require installation of another emission control device to reduce particulate emissions. Rather, the intent was to consider the fluid catalytic cracking unit catalyst regenerator together with any associated incinerator waste-heat boiler in terms of controlling particulate emissions. If an incinerator waste-heat boiler which burns additional liquid or solid fossile fuel is in use, an increase in total particulate emissions would be permitted, consistent with good control of particulate emissions from the combustion of that increment of fuel. Accordingly, fluid catalytic cracking unit incinerator waste-heat boilers have been deleted from the list of affected facilities. This amendment of Subpart J, however, is not a change in the intent of the existing standard of performance limiting emissions of particulate matter.

Public Participation

As prescribed by section 111 of the Clean Air Act as amended, this proposal of standards of performance has been preceded by the Administrator's determination that refinery sulfur recovery plants contribute significantly to air pollution which causes or contributes to the endangerment of public health or welfare. By publication of this determination in this issue of the Federal Register he adds petroleum refinery sulfur recovery plants to the list of affected facilities under the source category of petroleum refineries. In accordance with section 117 of the Act, publication of these proposed standards was preceded by

consultation with appropriate advisory committees, independent experts, and Federal departments and agencies.

Interested persons may participate in this rulemaking by submitting written comments (in triplicate) to the Emission Standards and Engineering Division, U.S. Environmental Protection Agency, Research Triangle Park, North Carolina 27711, Attention: Mr. Don R. Goodwin. The Administrator will welcome comments on all aspects of the proposed regulations, including the designation of sulfur recovery plants as a significant contributor to air pollution which causes or contributes to the endangerment of public health or welfare, economic and technological issues, and the proposed test method.

Comments are invited specifically on the severity of the economic impact of the proposed standards on the small refiner, since a number of interested parties have expressed objection to the absence of a lower level cut-off exempting the small refiner from compliance with the standards. Any comments submitted to the Administrator on this issue, however, should contain specific information and data pertinent to an evaluation of the magnitude of this impact and its severity.

Also, the Administrator would be especially interested in receiving comments regarding the issue of controlling reduced sulfur compound emissions without implementing section 111(d), particularly as this would affect the seven States involved.

All relevant comments received on or before December 3, 1976 will be considered. Comments received will be available for public inspection and copying at the EPA Public Information Reference Unit, Room 2922 (EPA Library), 401 M Street, SW., Washington, D.C. 20460.

Background information on these proposed standards of performance has been published in a document "Standard Support and Environmental Impact Statement—Standards of Performance for Petroleum Refinery Sulfur Plants." This document presents the factors considered in the development of the proposed standards, including alternative emission control systems, emission test data, environmental impact, costs and economic considerations. Copies of the document are available free of charge by writing to the Public Information Center (PM-215), U.S. Environmental Protection Agency, Washington, D.C. 20460 (specify "Standards Support and Environmental Impact Statement—Standards of Performance for Petroleum Refinery Sulfur Plants").

Authority. This notice of proposed rulemaking is issued under the authority of sections 111, 114 and 301(a) of the Clean Air Act, as amended (42 U.S.C. 1857c-6 and 9).

Dated: September 16, 1976.

JOHN QUARLES,
Acting Administrator.

It is proposed to amend 40 CFR Part 60 as follows:

Subpart J—Standards of Performance for Petroleum Refineries

1. Section 60.100 is revised as follows:

§ 60.100 Applicability and designation of affected facility.

The provisions of this subpart are applicable to the following affected facilities in petroleum refineries: fluid catalytic cracking unit catalyst regenerators, fuel gas combustion devices, and sulfur recovery plants.

2. Section 60.101 is amended by adding paragraphs (i) through (l) as follows:

§ 60.101 Definitions.

* * * * *

(i) "Sulfur recovery plant" means a process unit which converts hydrogen sulfide produced within a petroleum refinery to elemental sulfur. The sulfur recovery plant need not be physically located within a petroleum refinery.

(j) "Oxidation control system" means an emission control system which reduces emissions from sulfur recovery plants by converting these emissions to sulfur dioxide.

(k) "Reduction control system" means an emission control system which reduces emissions from sulfur recovery plants by converting these emissions to sulfur dioxide.

(k) "Reduction control system" means an emission control system which reduces emissions from sulfur recovery plants by converting these emissions to hydrogen sulfide.

(l) "Reduced sulfur compounds" mean hydrogen sulfide (H_2S), carbonyl sulfide (COS) and carbon disulfide (CS_2).

3. Section 60.102 is amended by revising paragraph (a) introductory text and paragraph (b) as follows:

§ 60.102 Standard for particulate matter.

(a) On and after the date on which the performance test required to be conducted by § 60.8 is completed, no owner or operator subject to the provisions of this subpart shall discharge or cause the discharge into the atmosphere from any fluid catalytic cracking unit catalyst regenerator:

* * * * *

(b) Where the gases discharged by the fluid catalytic cracking unit catalyst regenerator pass through an incinerator or waste heat boiler in which auxiliary or supplemental liquid or solid fossil fuel is burned, particulate matter in excess of that permitted by paragraph (a)(1) of this section may be emited to the atmosphere, except that the incremental rate of particulate matter emissions shall not exceed 43.0 g/MJ (0.10 lb/million Btu) of heat input attributable to such liquid or solid fossil fuel.

4. Section 60.104 is amended by revising paragraph (a) as follows:

§ 60.104 Standard for sulfur dioxide.

(a) On and after the date on which the performance test required to be conducted by § 60.8 is completed, no owner or operator subject to the provisions of this subpart shall:

(1) Burn in any fuel gas combustion device any fuel gas which contains hydrogen sulfide in excess of 230 mg/dscm (0.10 gr/dscf), except that the gases resulting from the combustion of fuel gas may be treated to control sulfur dioxide emissions provided the owner or operator demonstrates to the satisfaction of the Administrator that this is as effective in preventing sulfur dioxide emissions to the atmosphere.

(2) Discharge or cause the discharge of any gases into the atmosphere from any sulfur recovery plant containing in excess of:

(i) 0.025 percent by volume of sulfur dioxide at zero percent oxygen on a dry basis if emissions are controlled by an oxidation control system, or a reduction control system followed by incineration, or

(ii) 0.030 percent by volume of reduced sulfur compounds and 0.0010 percent by volume of hydrogen sulfide calculated as sulfur dioxide at zero percent oxygen on a dry basis if emissions are controlled by a reduction control system not followed by incineration.

* * * * *

5. Section 60.105 is amended by adding paragraphs (a) (4) and (5) and (e) (3) (i) and (ii) as follows:

§ 60.105 Emission monitoring.

(a) * * *

(4) An instrument for continuously monitoring and recording concentrations of H_2S in fuel gases burned in any fuel gas combustion device, if compliance with § 60.104(a) is achieved by removing H_2S from the fuel gas before it is burned; or an instrument for continuously monitoring and recording concentrations of SO_2 in the gases discharged into the atmosphere from the combustion of any fuel gas, if compliance with § 60.104(a) is achieved by removing SO_2 from the combusted fuel gases. If the concentration of H_2S is monitored, fuel gas combustion devices having a common source of fuel gas may be monitored at one location, if monitoring at this location accurately represents the concentration of H_2S in the fuel gas burned.

(5) An instrument for continuously monitoring and recording concentrations of SO_2 in the gases discharged into the atmosphere from the sulfur recovery plant if compliance with § 60.104(b) is achieved through the use of an oxidation control system, or a reduction control system followed by incineration; or an instrument for continuously monitoring and recording concentrations of H_2S and reduced sulfur compounds in the gases discharged into the atmosphere from the sulfur recovery plant if compliance with § 60.104(b) is achieved through the use

of a reduction control system not followed by incineration.

* * * * *

(e) * * *

(1) [Reserved]

(2) [Reserved]

(3) Sulfur dioxide. (i) Any hourly period during which the average concentration of H_2S in fuel gas combusted in any fuel gas combustion device subject to § 60.104(a) exceeds 230 mg/dscm (0.10 gr/dscf), if compliance with § 60.104(a) is achieved by removing H_2S from the fuel gas before it is burned; or any hourly period during which the average concentration of SO_2 in the gases discharged into the atmosphere from any fuel gas combustion device subject to § 60.104(a) exceeds the level specified in § 60.104(a), if compliance with § 60.104(a) is achieved by removing SO_2 from the combusted fuel gases.

(ii) Any four-hour period during which the average concentration of SO_2 in the gases discharged into the atmosphere from any sulfur recovery plant subject to § 60.104(b) exceeds the level specified in § 60.104(b)(1) if compliance with § 60.104(b) is achieved through the use of an oxidation control system or a reduction control system followed by incineration; or any four-hour period during which the average concentration of H_2S, or reduced sulfur compounds in the gases discharged into the atmosphere from any sulfur plant subject to § 60.104(b) exceeds the levels specified in § 60.104(b)(2) if compliance with § 60.104(b) is achieved through the use of reduction control systems not followed by incineration.

6. Section 60.106 is amended by revising paragraphs (c) and (d) as follows:

§ 60.106 Test methods and procedures.

* * * * *

(c) For the purpose of determining compliance with § 60.104(a), Method 11 shall be used to determine the concentration of H_2S and Method 6 shall be used to determine the concentration of SO_2.

(1) If Method 11 is used, the gases sampled shall be introduced into the sampling train at approximately atmospheric pressure. Where refinery fuel gas lines are operating at pressures substantially above atmosphere, this may be accomplished with a flow control valve. If the line pressure is high enough to operate the sampling train without a vacuum pump, the pump may be eliminated from the sampling train. The sample shall be drawn from a point near the centroid of the fuel gas line. The minimum sampling time shall be 10 minutes and the minimum sampling volume 0.01 dscm (0.35 dscf) for each sample. The arithmetic average of two samples of equal sampling time shall constitute one run. Samples shall be taken at approximately 1-hour intervals. For most fuel gases, sample times exceeding 20 minutes may result in depletion of the collecting solution, although fuel gases containing low concentrations of hydrogen sulfide may necessitate sampling for longer periods of time.

(2) If Method 5 is used, Method 1 shall be used for velocity traverses and Method 2 for determining velocity and volumetric flow rate. The sampling site for determining SO_2 concentration by Method 6 shall be the same as for determining volumetric flow rate by Method 2. The sampling point in the duct for determining SO_2 concentration by Method 6 shall be at the centroid of the cross section if the cross sectional area is less than 5 m^2 (54 ft^2) or at a point no closer to the walls than 1 m (30 inches) if the cross sectional area is 5 m^2 or more and the centroid is more than one meter from the wall. The sample shall be extracted at a rate proportional to the gas velocity at the sampling point. The minimum sampling time shall be 10 minutes and the minimum sampling volume 0.01 dscm (0.36 dscf) for each sample. The arithmetic average of two samples of equal sampling time shall constitute one run. Samples shall be taken at approximately one-hour intervals.

(d) For the purpose of determining compliance with § 60.104(b), Method 6 shall be used to determine the concentration of SO_2 and Method 15 shall be used to determine the concentration of H_2S and reduced sulfur compounds.

(1) If Method 6 is used, the procedure outlined in paragraph (c)(2) of this section shall be followed except that each run shall span a minimum of four consecutive hours of continuous sampling. A number of separate samples may be taken for each run, provided the total sampling time of these samples adds up to a minimum of four consecutive hours. Where more than one sample is used, the average SO_2 concentration for the run shall be calculated as the time weighted average of the SO_2 concentration for each sample according to the formula:

$$C_R = \sum_{i=1}^{N} C_{s_i} \frac{t_{s_i}}{T}$$

Where:

C_R = SO_2 concentration for the run.

N = Number of samples.

C_{s_i} = SO_2 concentration for sample i.

t_{s_i} = Continuous sampling time of sample i.

T = Total continuous sampling time of all N samples.

(2) If Method 15 is used, each run shall consist of 16 samples taken over a minimum of four hours. The sampling point shall be at the centroid of the cross section of the duct if the cross sectional area is less than 5 m^2 (54 ft^2) or at a point no closer to the walls than 1 m (30 inches) if the cross sectional area is 5 m^2 or more and the centroid is more than 1 meter from the wall. To insure minimum residence time for the sample inside the sample lines, the sampling rate shall be at least 3 liters/minute (0.1 ft^3/min). The SO_2 equivalent for each run shall be calculated as the arithmetic average of the SO_2 equivalent of each sample during the run. Reference Method 4 shall be used to determine the moisture content of the gases. The sampling point for Method 4 shall be adjacent to the sampling point for Method 15. The sample shall be extracted at a rate proportional to the gas velocity at the sampling point. Each run shall span a minimum of four consecutive hours of continuous sampling. A number of separate samples may be taken for each run provided the total sampling time of these samples adds up to a minimum of four consecutive hours. Where more than one sample is used, the average moisture content for the run shall be calculated as the time weighted average of the moisture content of each sample according to the formula:

$$B_{wo} = \sum_{i=1}^{N} B_{si} \left[\frac{t_{si}}{T}\right]$$

B_{wo} = Proportion by volume of water vapor in the gas stream for the run.

N = Number of samples.

B_{si} = Proportion by volume of water vapor in the gas stream for the sample i.

t_{si} = Continuous sampling time for sample i.

T = Total continuous sampling time of all N samples.

* * * * *

APPENDIX A—REFERENCE METHODS

7. Appendix A is amended by adding a new reference method as follows:

METHOD 15—DETERMINATION OF HYDROGEN SULFIDE, CARBONYL SULFIDE, AND CARBON DISULFIDE EMISSIONS FROM STATIONARY SOURCES

1. *Principle and applicability.* 1.1 Principle. A gas sample is extracted from the emission source and diluted with clean dry air. An aliquot of the diluted sample is then analyzed for hydrogen sulfide (H_2S), carbonyl sulfide (COS), and carbon disulfide (CS_2) by gas chromatographic (GC) separation and flame photometric (FPS) detection.

1.2 Applicability. This method is applicable for determination of hydrogen sulfide, carbonyl sulfide, and carbon disulfide when specified by an applicable subpart.

2. *Range and sensitivity.* 2.1 Range. The maximum limit of the FPD for each sulfur compound is dependent on the sample size. For a 1 ml sample the upper limit is about 10 ppm. It may be necessary to dilute gas samples from sulfur recovery plants hundredfold (100:1), resulting in an upper limit of about 1000 ppm for each compound.

2.2 The minimum detectable concentration of the FPD is also dependent on sample size and would be about 0.5 ppm for a 1 ml sample.

3. *Interferences.* 3.1 Moisture Condensation. Moisture condensation in the sample delivery system, the analytical column, or the FPD burner block can cause losses or interferences. This potential is eliminated by heating the sample line, and by conditioning the sample with dry dilution air to lower its dew point below the operating temperature of the GC/FPD analytical system prior to analysis.

3.2 Carbon Monoxide and Carbon Dioxide. CO and CO_2 have a substantial densensitizing effect on the detector even after 10:1 dilution. This potential interferenc is eliminated by eluting CO and CO_2 with the "air peak" prior to elution of any sulfur compound.

3.3 Elemental Sulfur. The condensation of sulfur vapor in the sampling lines can lead to eventual coating and even blockage of the sample line. This problem can be eliminated along with the moisture problem by heating the sample line.

4. *Precision.* 4.1 Calibration Precision. A series of three consecutive injections of the same calibration gas, at any dilution, should product results which do not vary by more than ±3% from the mean of the three injection.

4.2 Calibration Drift. The calibration drift determined from the mean of three injections made at the beginning and end of any 8-hour period should not exceed ±3%.

5. *Sample apparatus.* 5.1.1 Sample probe—The sample probe should be fabricated from a suitable length and diameter of stainless steel tubing.

5.1.2 Sample line—0.48 cm (3/16 inch) inside diameter Dekoron FEP Teflon[1] tubing, heated to 120° C. This temperature is controlled by a thermostatic heater.

5.1.3 Sample pump—Leakless Teflon coated diaphragm type or equivalent. The pump head is heated to 120° C by enclosing it in the sample dilution box (5.2.4 below).

[1] Mention of trade names or specific products does not constitute an endorsement by the Environmental Protection Agency.

5.2 Dilution System. A schematic diagram of the dynamic dilution system is given in Figure 15–1. The dilution system is constructed such that all sample contacts are made of inert materials. The dilution system which is heated to 120° C must be capable of a minimum of 10:1 dilution of sample. Equipment used in the dilution system is listed below.

5.2.1 Dilution pump—Model A–150 Komhyr Teflon positive displacement type, non-adjustable 150 cc/min±2.0%, or equivalent, per dilution stage. A 10:1 dilution of sample is accomplished by combining 150 cc of sample with 1350 cc of clean dry air as shown in Figure 15–1.

5.2.2 Valves—Threeway Teflon solenoid or manual type.

5.2.3 Tubing—Teflon tubing and fittings are used throughout from the sample probe to the GC/FPD to present an inert surface for sample gas.

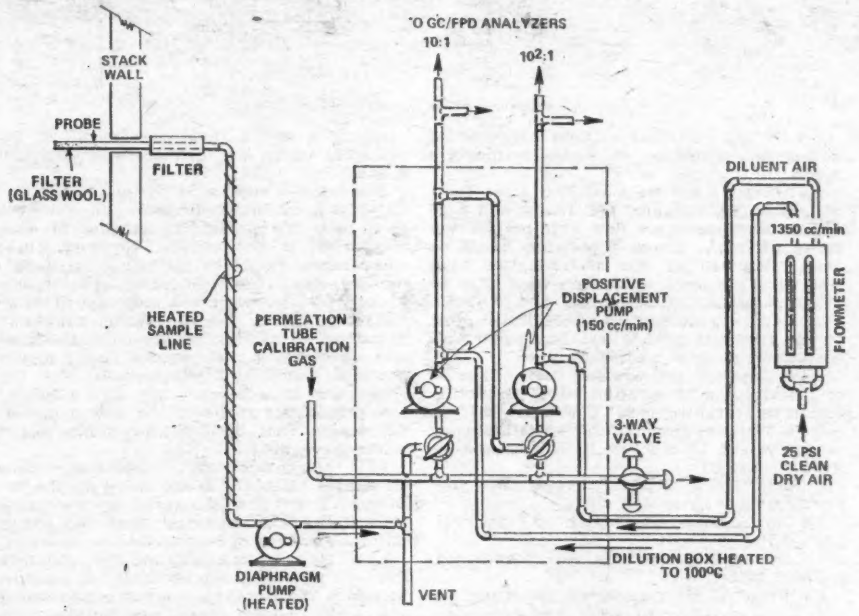

Figure 15.1. Sampling and Dilution Apparatus.

5.2.4 Box — Insulated box, heated and maintained at 120° C, of sufficient dimensions to house dilution apparatus.

5.2.5 Flowmeters—Rotameters or equivalent to measure flow from 0 to 1500 ml/min ±1% per dilution stage.

5.3 Gas Chromatograph.

5.3.1 Column—1.83 m (6 ft) length of Teflon tubing, 2.16 mm (0.085 in.) inside diameter, packed with deactivated silica gel, or equivalent.

5.3.2 Sample valve—Teflon six-port gas sampling valve equipped with a 10 ml sample loop, actuated by compressed air (Figure 15–2).

5.3.3 Oven—For containing sample valve, stripper column and separation column. The oven should be capable of maintaining an elevated temperature ranging from ambient to 100° C, constant with ±1° C.

5.3.4 Temperature monitor — Thermocouplepyrometer to measure column oven, detector, and exhaust temperature ±1° C.

5.3.5 Flow system—Gas metering system to measure sample flow, hydrogen flow, oxygen flow and nitrogen carrier gas flow.

5.3.6 5.3.6 Detector—Flame photometric detector.

5.3.7 Electrometer—Capable of full scale amplification of linear ranges of 10^{-9} to 10^{-4} amperes full scale.

5.3.8 Power supply—Capable of delivering up to 750 volts.

5.3.9 Recorder—Compatible with the output voltage range of the electrometer.

5.4 Calibration. Permeation tube system (Figure 15–3).

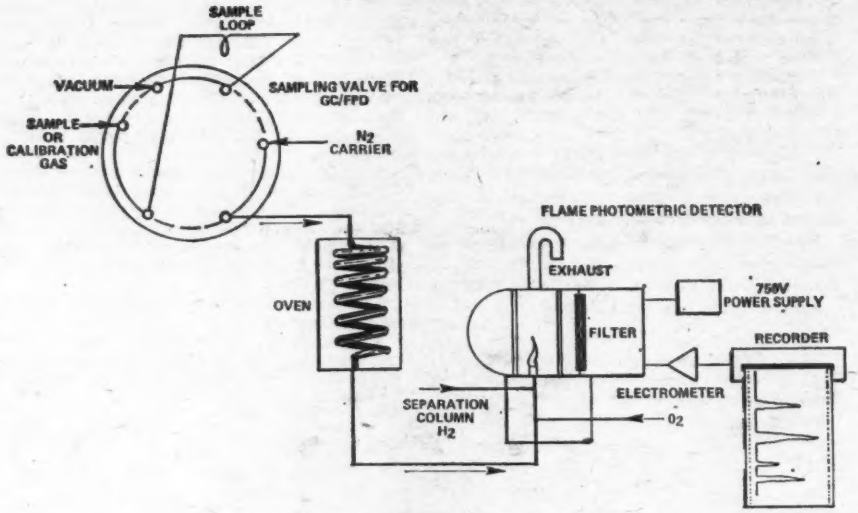

Figure 15.2. Gas Chromatographic Flame Photometric Analyzer.

5.4.1 Tube chamber — Glass chamber of sufficient dimensions to house permeation tubes.

5.4.2 Mass flowmeters — Two mass flowmeters in the range of 0–3 l/min and 0–10 l/min to measure air flow over permeation tubes at ±2%. These flowmeters shall be cross-calibrated at the beginning of each test with a known calibration gas (H_2S or other), comparing responses by GC/FPD. Each mass flowmeter shall be calibrated prior to the first test with a wet test meter and thereafter, at least once each year.

5.4.3 Constant temperature bath—Capable of maintaining permeation tubes at certification temperature at 30° C within ±0.1° C.

5.4.4 Temperature monitor—Thermometer or equivalent to monitor bath temperature within ±0.1° C.

6. *Reagents.* 6.1 Fuel. Hydrogen (H_2) prepurified grade or better.

6.2 Combustion Gas. Oxygen (O_2) prepurified grade or better.

6.3 Carrier Gas. Nitrogen (N_2) prepurified grade or better.

6.4 Diluent. Air containing less than 0.5 ppb total sulfur compounds and less than 10 ppm each of moisture and total hydrocarbons, and filtered using MSA filters 46727 and 79030 or equivalent. Removal of sulfur compounds can be verified by injecting dilution air only, described in Section 8.4.

6.5 Compressed Air. 60 psig for GC valve actuation.

6.6 Calibration Gases. Permeation tubes gravimetrically calibrated and certified at 30.0° C.

7. *Pre-test procedures.* 7.1 After the complete measurement system has been set up at the site and verified to be operational, the following procedures shall be completed before sampling is initiated.

7.1.1 Flowmeter calibration and leak test—Appropriate calibration and leak test procedures shall be employed to verify the accuracy and/or integrity of all flowmeters, sample lines, and connections. For components under negative presure (lines and connections upstream of the sample pump), a leak test is performed using the sample pump to pull a vacuum. Attach the probe end of the sample line to a manometer or vacuum gauge, start the pump and pull greater than 50 mm (2 in.) Hg vacuum. Close off the pump outlet, then stop the pump and ascertain that there is no leak for 5 minutes. For components subject to positive pressure, leak tests shall be performed at each joint by applying a liquid (detergent in water, for example) which will foam in the presence of a leak.

Since the complete dilution system and GC/FPD is calibrated prior to and following each test, the precise calibration of each component is not critical. However, these components should be verified to be operating properly. This verification can be performed by observing the response of flowmeters or of the GC output to changes in flow rates or calibration gas concentrations and ascertaining the response to be within predicted limits. If any component, or if the complete system fails to respond in a normal and predictable manner, the source of the discrepancy shall be identified and corrected before proceeding.

7.1.2 Mass flowmeter calibration—Since all sample calculations are based on the accuracy of air flow measured by the mass flowmeters, it is essential that the cross-calibration check of Section 5.4.2 be followed.

7.1.3 Prior to any sampling run, calibrate the system using the procedures set forth in Section 8. If more than one run is performed during any 24-hour period, a calibration need not be performed prior to the second and any subsequent runs. The calibration must, however, be verified as prescribed in Section 8, after the last run made within the 24-hour period.

8. *Calibration.* 8.1 General Considerations. This section outlines steps to be followed for use of the GC/FPD and the dilution system. The procedure does not include detailed instructions because operation of these systems is complex, and it requires an understanding of the individual system being used. Each system should include a written operating manual describing in detail the operating procedures associated with each component in the measurement system. In addition, the operator should be familiar with the operating principles of the components, particularly the GC/FPD. The references cited at the end of this method are recommended for review for this purpose.

8.2 Permeation Tubes. Under the following procedure, accurately known concentrations (±1%) of a variety of sulfur compounds are generated by passing clean dry air or other diluent gas over permeation tubes, each containing a specific sulfur compound as a permeant. These tubes consist of hermetically sealed FEP Teflon tubing in which a liquefied gaseous substance is enclosed. The enclosed gas permeates through the tubing wall

at a constant rate. When the temperature is constant, calibration gases covering a wide range of known concentrations can be generated by varying and accurately measuring the flow rate of diluent gas passing over the tubes. These calibration gases are used to calibrate the GC/FPD system and the dilution system.

8.3 Calibration Procedure. Assemble the permeation tube calibration apparatus as depicted in Figure 15–3. Insert the permeation tubes into the glass tube chamber. Check the bath temperature to assure agreement with the calibration temperature of the tubes within ±1° C. A temperature of 30° C is recommended for the sulfur gas tubes. Allow several hours for the tubes to equilibrate. When equilibrated, vary the flow rate of diluent air flowing over the tubes to produce the desired concentrations for calibrating the analytical and dilution systems. The air flow across the tubes must at all times exceed the flow requirement of the analytical systems. The concentration in parts per million generated by a tube containing a specific permeant can be calculated as follows:

Equation 15-1

$$C = K\frac{P_r}{ML}$$

Where:
C = Concentration of permeant produced in ppm.
P_r = Permeation rate of the tube in μg/min.
M = Molecular weight of the permeant $\left(\frac{g}{g\text{-mole}}\right)$.
L = Flow rate, l/min, of air over permeant @20° C, 760 mm Hg.
K = Gas constant at 20° C and 760 mm Hg = 24.04 l/g mole.

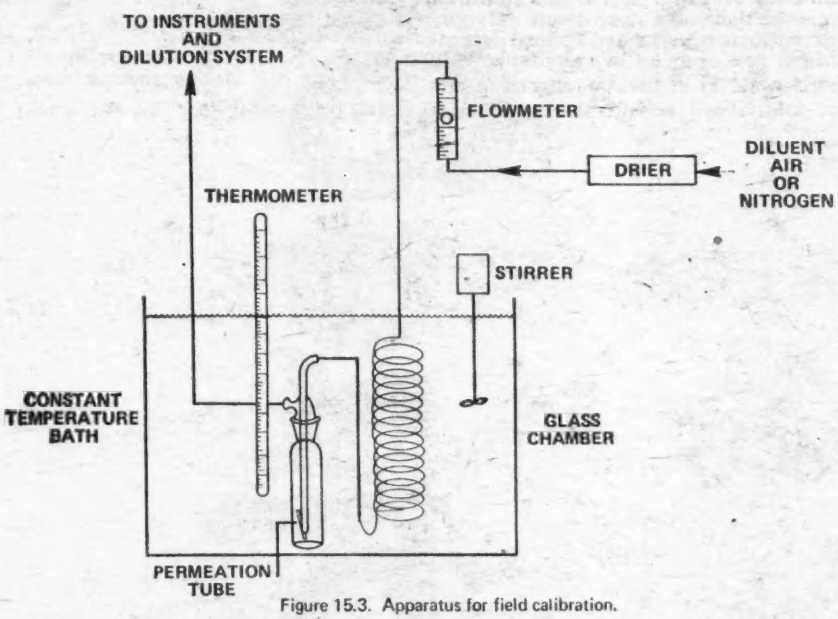

Figure 15.3. Apparatus for field calibration.

8.4 Operating Condition for GC/FPD System. The operation parameters for the GC/FPD system are as follows: nitrogen carrier gas flow rate of 100 cc/minute, exhaust temperature of 110° C, detector temperature of 105° C, oven temperature of 40° C, hydrogen flow rate of 80 cc/minute, oxygen flow rate of 20 cc/minute, and sample flow rate of 80 cc/minute.

8.5 Calibration of GC/FPD Analysis Systems. Generate a series of three or more known concentrations spanning the linear range of the RPD (approximately 0.05 to 1.0 ppm) for each sulfur compound anticipated to be present in the gas stream analyzed. Bypassing the dilution system, inject these standards into the GC/FPD analyzers and monitor the responses. Three injects for each concentration must yield the precision described in Section 4.1. Failure to attain this precision is an indication of a problem in the calibration or analytical system. Any such problem must be identified and corrected before proceeding. Peak heights, rather than integrated areas, have proven satisfactory; however, integrated areas may be required for alternate columns or instrumentation.

8.5.1 Calibration curves—Plot the GC/FPD responses in current (amperes) versus their causative concentrations in ppm on log-log coordinate graph paper for each sulfur compound calibrated.

8.6 Calibration of Dilution System. Generate a known concentration of hydrogen sulfide using the permeation tube system. Adjust the flow rate of diluent air for the first dilution stage so that the desired level of dilution is approximated. Inject the diluted calibration gas into the GC/FPD and monitor its response. Three injects for each dilution must yield the precision described in Section 4.1. Failure to attain this precision in this step is an indication of a problem in the dilution system. Any such problem must be identified and corrected before proceeding. Using the calibration curve for H_2S (developed under 8.5.1), determine the diluted calibration gas concentration in ppm. Then, calculate the dilution factor as the ratio of the calibration gas concentration before dilution to the diluted calibration gas concentration determined under this paragraph. Repeat this procedure for each stage of dilution required.

9. *Sampling and analysis procedure.*

9.1 Sampling. Insert the sampling probe into the test port making certain that no dilution air enters the stack through the port. Begin sampling and dilute the sample approximately 10:1 using the dilution system shown in Figure 15–1. Note that the precise dilution factor is that which is determined in paragraph 8.6. Condition the entire system with sample for a minimum of 15 minutes prior to commencing analysis.

9.2 Analysis. The sample value is actuated for one minute in which time an aliquot of diluted sample is injected onto separation column. The valve is then deactivated for the remainder of the analysis cycle in which time the sample loop is refilled and the separation column continues to be flushed. The elution time for each compound will be determined during calibration.

9.3 Sample Run A sample consists of one analysis (injection).

10. *Post-test procedures.*

10.1 Recalibration. After each run, or after a series of runs made within a 24-hour period, perform a partial recalibration using the procedures in Section 8. Only H_2S (or other permeant) need be used to recalibrate the GC/FPD analysis system (8.4) and the dilution system (8.5).

10.2 Determination of Calibration Drift. Compare the calibration curves obtained prior to the runs to the calibration curves obtained under paragraph 10.1. The calibration drift must not exceed the limits set forth in paragraph 4.2. If the drift exceeds this limit, the intervening run or runs shall be considered not valid.

11. *Calculations.*

11.1 Determine the concentrations of hydrogen sulfide, carbonyl sulfide, and carbon disulfide detected in each sample directly from the calibration curves.

11.2 Sulfur Dioxide Equivalent. For each sample the concentrations of hydrogen sulfide, carbonyl sulfide, and carbon disulfide are totaled and the sum expressed as sulfur dioxide using the following equation:

$$SO_2 \text{ equivalent} = [H_2S] + [COS] + 2[CS_2]$$

SO_2 equivalent = The sum of the concentration of each of the measured compounds (H_2S, COS, CS_2) expressed as sulfur dioxide, in ppm.
$[H_2S]$ = Measured concentration of hydrogen sulfide, in ppm.
$[COS]$ = Measured concentration of carbonyl sulfide, in ppm.
$[CS_2]$ = Measured concentration of CS_2, in ppm.

12. *Bibliography.*

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ENVIRONMENTAL PROTECTION AGENCY

[FRL 610-5]

AIR POLLUTION PREVENTION AND CONTROL

Addition to the List of Categories of Stationary Sources

Section 111 of the Clean Air Act (42 U.S.C. 1857c-6) directs the Administrator of the Environmental Protection Agency to publish, and from time to time revise, a list of categories of stationary sources which he determines may contribute significantly to air pollution which causes or contributes to the endangered of public health or welfare. Within 120 days after the inclusion of a category of stationary sources in such list, the Administrator is required to propose regulations establishing standards of performance for new and modified sources within such category. At present, standards of performance for 24 categories of sources have been promulgated.

The Administrator, after evaluating available information, has determined that petroleum refinery sulfur recovery plants are an additional facility within the petroleum refinery category which meets the above requirements. The basis for this determination is discussed in the preamble to the proposed regulation that is published at page 43866 in this issue of the FEDERAL REGISTER. Evaluation of other stationary source categories is in progress, and the list will be revised from time to time as the Administrator deems appropriate. Accordingly, notice is given that the Administrator, pursuant to section 111(b)(1)(A) of the Act and after consultation with appropriate advisory committees, experts, and Federal departments and agencies in accordance with section 117(f) of the Act, effective (date of publication) amends the list of categories of stationary sources to read as follows:

LIST OF CATEGORIES OF STATIONARY SOURCES AND CORRESPONDING AFFECTED FACILITIES

Source category	*Affected facilities*
* * * * *	
7. Petroleum refineries.	Fluid catalytic cracking unit catalyst regenerators. Fuel Gas Combustion Devices. Sulfur Recovery Plants.
* * * * *	

Proposed standards of performance applicable to the above source category appear at page 43866 in this issue of the FEDERAL REGISTER.

Dated: September 16, 1976.

JOHN QUARLES,
Acting Administrator.

[FR Doc.76-28710 Filed 10-1-76;8:45 am]